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Prototype Thin-Film Thermocouple/ Heat-Flux Sensor for a Ceramic-Insulated Diesel Engine

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SUMMARY

A platinum versus platinum-13 percent rhodium thin-film thermocouple/heat-flux sensor was devised and tested in the harsh, high-temperature environment of a ceramic-insulated, low-heat-rejection diesel engine. The sensor probe assembly was developed to provide experimental validation of heat transfer and thermal analysis methodologies applicable to the insulated diesel engine concept. The thin-film thermocouple configuration was chosen to approximate an uninterrupted chamber surface and provide a one dimension heat-flux path through the probe body. The engine test was conducted by Purdue University for Integral Technologies Inc., under a DOE-funded contract managed by NASA Lewis Research Center. The thin-film sensor performed reliably during 6 to 10 hr of repeated engine runs at indicated mean surface temperatures up to 950 K. However, the sensor suffered partial loss of adhesion in the thin-film thermocouple junction area following maximum cyclic temperature excursions to greater than 1150 K.

INTRODUCTION

Experimental verification of heat transfer and thermal analysis modeling methodologies as applied to the insulated diesel engine is very difficult due to a lack of suitable instrumentation for obtaining accurate measurements of surface temperature at operating conditions. A basic difficulty is that the thermocouple temperature sensors and heat-flux gauges must be small and nonintrusive. In addition, the heat-flux gauge must function under transient conditions. Reliable measurements under operating conditions are essential to the development of dependable design methods applicable to ceramic-insulated, low-heat-rejection engines.

Use of thin films of material to form thermocouples has been pursued for a wide variety of applications from measurement of gun bore surface temperatures (ref. 1) to high temperature measurements in jet aircraft gas turbines (ref. 2). An early investigation of sputtered thin-film thermocouples was conducted for application on jet engine turbine blades (ref. 3). This work demonstrated that the thin-film thermocouple could accurately record high steady-state metal temperatures to 1020 K with potential to higher temperatures. More recently, thin-film thermocouples were developed for surface temperature measurement of air-cooled turbine blades (refs. 4 to 7). Although thermocouples in wire form are commonly used in heat-flux measuring devices, the application of thin-film materials has been investigated in an experiment involving a flat, stainless steel plate with sputtered nickel film and base metal forming the thermocouple hot junction (ref. 8).

In the subject investigation, a prototype heat-flux sensor was fabricated by installing an embedded wire thermocouple in an iron plug coated with yttria-stabilized zirconia on which the thin-film thermocouple was sputter deposited. The thermoelement legs of platinum and platinum-13 percent rhodium were used for both the sputtered and embedded wire thermocouples. The design constraints were that the heat-flux sensor must fit in the existing access ports, resolve fluctuation in heat flux, and survive engine environment with surface temperatures up to 1000 K.

During a test period from January through April 1987, the prototype thin-film thermocouple/heat-flux sensor was used in a ceramic-insulated, single cylinder, diesel engine test program at Purdue University by Integral Technologies Inc., under a DOE-funded contract managed by NASA Lewis Research Center. The test program was designed to provide experimental validation of heat transfer and thermal analysis modeling methodologies applicable to the low-heat-rejection (LHR) diesel engine concept. Firedeck surface temperature and heat-flux measurements were made in both insulated and uninsulated engine configurations. Commercially available metal heat-flux sensors were used for comparison only during baseline engine tests.

PROTOTYPE SENSOR FABRICATION

The prototype sensor assembly was fabricated by coating an iron plug with yttria-stabilized zirconia onto which the thin-film thermocouple was sputter deposited. During the process, procedures were established for surface preparation, sputter deposition of a thin-film thermocouple that would adhere well to the ceramic surface of the sensor probe, and attachment of leadwires to the thin film. A wire thermocouple was embedded in the iron plug to form the cold-side thermocouple. The thermoelement legs of platinum and platinum-13 percent rhodium were used for both the sputtered and embedded wire thermocouples, in consideration of the expected hostile environment and surface temperatures exceeding 1000 K.

The iron plug, as shown in figure 1, was coated with a 1.25 mm (0.050 in.) thick layer of yttria-stabilized zirconia over the MCrAlY bond coat using a commercial plasma spraying process. The as-sprayed zirconia surface was, then, chemically cleaned in ultrasonic baths for 10 min in each of the following solvents, in order: acetone, isooctane, methylene chloride, and ethyl alcohol. After the chemical cleaning, the iron plug was baked in an oven at 450 K for 2 hr. Further cleaning to remove any residual organics was completed by plasma etching the zirconia for 30 min at 200 W power level with a plasma gas of argon with 10 percent oxygen. An electrically insulating aluminum oxide bond coat was then sputtered to a thickness of 2 μm on the unpolished zirconia surface. Previous measurements made in this laboratory indicated that the resistivity of aluminum oxide film was greater than 10^7 Ω-cm in the range of 600 to 1250 K.

The thin-film thermocouple was sputtered on the aluminum oxide layer using a metal mask. The thickness of the sputtered film was monitored using a glass slide placed in the sputtering chamber along side the actual specimen being sputtered. A portion of the glass slide was masked with a thin cover glass to create a step change in contour which can be measured with a profilometer. As shown in figure 2 the aluminum oxide insulating layer was ~2 μm thick, platinum was 5 μm thick, and platinum-13 percent rhodium was 3 μm thick.

The thermocouple junction was formed by slightly overlapping the circular films of platinum and platinum-13 percent rhodium as shown in figure 1. This geometry simplified mask preparation and provided sufficient surface area to make repairs in the event a leadwire became detached from the thin film. The excess surface provided adequate area to test for film adherence without contaminating the thermocouple junction zone. Further, the circular geometry was selected to minimize stress concentration sites and promote a more diffuse stress pattern in the sputtered thin film.

Table I lists the sputtering parameters associated with depositing the aluminum oxide, platinum, and platinum-13 percent rhodium thin films using radiofrequency sputtering equipment. Radiofrequency sputtering was selected for deposition of the aluminum oxide electrical insulation and thermocouple thin films because the process allows sputtering of both metallic alloys and ceramic materials. The adhesion of such sputtered films is generally better than films deposited by other physical vapor deposition processes such as electron beam deposition and plasma spraying. Furthermore, in the radiofrequency sputtering process, the original composition of materials to be deposited does not significantly change during the sputtering. As a consequence, the thermoelectric potential variation with temperature for the deposited thin film is expected to be similar to the source materials.

Figure 3 shows the elemental analysis of the platinum-13 percent rhodium film. The rhodium concentration in the sputtered film was 13.4 percent by weight percent determined by the energy-dispersive x-ray analysis of the sputtered film on a glass slide exposed during thin-film thermocouple deposition. Similar x-ray analysis of the platinum film sputtered on a glass slide showed that no other metallic element was present in significant quantity in the sputtered film. Figure 4 shows the surface of the sputtered thin film on a smooth glass substrate compared with an unpolished plasma sprayed zirconia substrate.

Swaged thermocouple leadwires were installed as shown on figure 1. The exposed ends, flattened to about 25 to 75 μm (0.001 to 0.003 in.), were welded onto the sputtered thin films using a parallel gap welder with constant voltage power supply. Representative parallel gap welding schedule parameters are listed in table II.

Figure 5 shows some results of early attempts to establish the final welding parameters. Figure 5(a) shows a mild weld in which no visible change in the wire is observed. Figure 5(b) shows a slight melting of the wire and figure 5(c) shows the shrinkage cavity resulting from more extensive melting of the metal. The figure 5(d) shows the burned metal and disruption of the thin film due to excessive heat. Acceptable welds, figures 5(b) and (c), were produced using the welding parameters listed in table II.

The initial functional test, not a calibration test, of the completed thin-film thermocouple and leadwire assembly was performed in air using a controlled temperature oven (fig. 6). Temperature readings from the thin-film thermocouple tracked readings from the exposed junction reference thermocouple during incremental temperature changes between ambient and 870 K. Sputtered films and leadwire weldments remained intact.

ENGINE TEST RESULTS

During January through April 1987, the prototype thin-film thermocouple/heat-flux sensor assembly was installed and tested in the ceramic-insulated, single cylinder, low-heat-rejection diesel engine located at Purdue University. This engine testing was conducted by Purdue University for Integral Technologies Inc., under a DOE-funded contract managed by NASA Lewis Research Center. Detailed results of the entire testing program, including performance characteristics of the sensor, are to be presented later in scheduled reports or research papers under the contract. However, for the purpose of this paper, the following brief description of the related portion of the engine test results is presented (ref. 9).

Measurements were made at two locations on the firedeck above the piston crown and bowl locations. Figure 7 shows the firedeck including the fuel injector on the cylinder centerline, dual intake and exhaust valve locations, and a sensor mounting port near the cylinder wall position which places the sensor over the piston crown. One of the intake valves on this engine was replaced with a plug so as to provide a sensor port location over the bowl portion of the top of the piston.

Analysis of initial heat transfer data taken during diesel engine tests indicated agreement with predicted levels of heat flux at indicated firedeck surface temperatures up to 950 K. This agreement with predicted heat-flux levels was found for both cooled and insulated engine configurations using the prototype probe in instrument ports above both the piston crown and bowl locations (fig. 7). Peak values of heat flux were found to be lower in the insulated engine configuration (fig. 8). Peak heat-flux values were determined from the difference between the maximum surface temperature on the probe and the temperature from the thermocouple embedded in the probe.

Approximately 40 percent of the intended engine test matrix was completed using two prototype sensor assemblies. These assemblies functioned reliably during 6 to 10 hr of repeated engine operations. The prototype heat-flux sensor survived engine test runs at indicated mean surface temperatures up to 950 K, speeds up to 2000 rpm, and loads up to 50 percent full load. However, the sensor delaminated following maximum cyclic temperature excursions to greater than 1150 K. Circled data points in figure 9 show the deviation from extrapolated temperature swing at the high fuel loadings. Subsequent recheck at the lower fuel loading still indicated higher than expected temperature swing. The temperature swing is the maximum temperature variation during the four stroke cycle at the point of measurement.

The sputtered thin film of platinum apparently blistered, whereas the film of platinum-13 percent rhodium showed no visible anomaly. A post-test examination by energy-dispersive x-ray spectroscopy indicated that the platinum film separated from the underlying aluminum oxide layer, in the form of many small, blister-like, raised areas as shown in figure 10. The underlying aluminum oxide layer adhered well to the zirconia substrate.

CONCLUDING REMARKS

A special heat-flux sensor with a surface deposited thin-film thermocouple was developed for use in the harsh, high temperature environment expected

in a ceramic-insulated, low-heat-rejection diesel engine. This prototype sensor consisted of embedded wire and sputter deposited thin-film thermocouples of type R. Thin films of platinum and platinum-13 percent rhodium were sputter deposited on the aluminum oxide insulated surface of a yttria-stabilized zirconia layer. The zirconia layer was deposited on the sensor plug by a commercially available plasma spraying process. The leadwires were attached to the sputtered thin films using a parallel gap welder.

The thin-film thermocouple configuration was chosen for minimal disturbance of boundary layer temperature and flow over the sensor plug and minimal effect on heat flux through the plug and fast time response. The prototype thin-film thermocouple/heat-flux sensor assembly was installed and tested in a ceramic-insulated, low-heat-rejection diesel engine at Purdue University. This engine testing was conducted by Purdue University for Integral Technologies Inc., under a DOE-funded contract managed by NASA Lewis Research Center. The prototype heat-flux sensor was tested to provide experimental validation of heat transfer and thermal analysis methodologies applicable to the ceramic-insulated, low-heat-rejection diesel engine concept.

The thin-film thermocouple/heat-flux sensor performed reliably during 6 to 10 hr of repeated engine runs at indicated mean surface temperatures up to 950 K. This sensor has survived engine test runs at speeds up to 2000 rpm and loads up to 50 percent full load. However, the sensor suffered partial loss of adhesion in the thin-film thermocouple junction area following maximum cyclic temperature excursions to greater than 1150 K. Efforts to improve adhesion of platinum film are underway.

There are four approaches identified for improving the adhesion of platinum and platinum-rhodium films deposited on the alumina surface (ref. 10). These approaches are based on the adhesion mechanisms of chemical and mechanical bonding between thin metal films and ceramic substrates.

(1) Deposition of a thin layer of Pt_xO_{1-x} cermet at the film-to-substrate interface.

(2) Deposition of a thin layer of a metal, such as chromium or molybdenum, at the Pt-Al_2O_3 interface.

(3) Application of a strong electric field from the metal film to the insulator at high temperature in order to enhance the polarity of the interface.

(4) Bombardment of the substrate with energetic ions.

Improvement in the adhesion of platinum film by depositing a Pt_xO_{1-x} interlayer at the film-to-substrate interface has been reported (ref. 11). A high degree of adhesion was obtained in the case of the platinum film which had a 300 Å thick Pt_xO_{1-x} at the aluminum oxide interface.

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thermocouple/heat-flux sensor for a ceramic-insulated diesel engine. We would also like to thank Charles E. Hulbert, Jr., for providing the energy-dispersive x-ray spectroscopic analysis of the delaminated thin-film thermocouple. We would also like to thank Purdue University, particularly Dale R. Tree of the School of Mechanical Engineering for providing the engine test results.

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TABLE I. - COMPARATIVE SPUTTERING PARAMETERS FOR THIN-FILM THERMOCOUPLE AND ALUMINUM OXIDE

	Aluminum oxide	Platinum	Platinum-13-percent rhodium
Method	RF magnetron	RF diode	RF diode
Cathode distance, cm	3.2	5.4	5.4
Baseline pressure, mm Hg	1.8×10^{-7}	1×10^{-7}	3.4×10^{-7}
Argon, mm Hg	9.5×10^{-3}	5×10^{-3}	5×10^{-3}
Oxygen, mm Hg	1.5×10^{-3}	------	--------
Forward power, W	600	250	250
Reflected power, W	5	<1	<1
Bias cap. setting	--------	35.7	28.1
Input cap. setting	6.5	6.5	0
Load tune setting	-2.5	-4	-6
Time, hr	5	4	3
Thickness, μm	1.7	4.8	3

TABLE II. - REPRESENTATIVE SCHEDULE FOR WELDING LEADWIRES TO THIN FILM

Wire diameter, μm (mil)	150 (6) flattened to 50 (2) prior to welding
Thin film thickness, μm	3 to 5
Parallel gap welder	
Power supply	Constant voltage
Voltage, V	1.10
Time, msec	10.0
Electrode	
Type	Molybdenum-base alloy
Gap, μm (mil)	125 (5)
Tip size, mm	0.635x1.14
Force, g	400

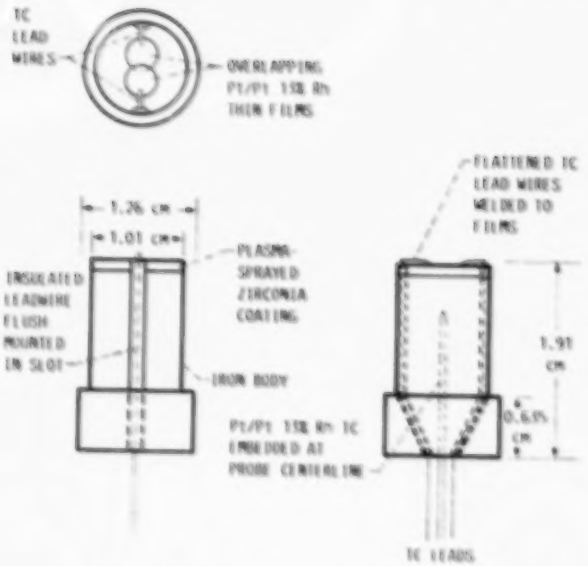


FIGURE 1. - INSULATED TOTAL HEAT FLUX PROBE ASSEMBLY.

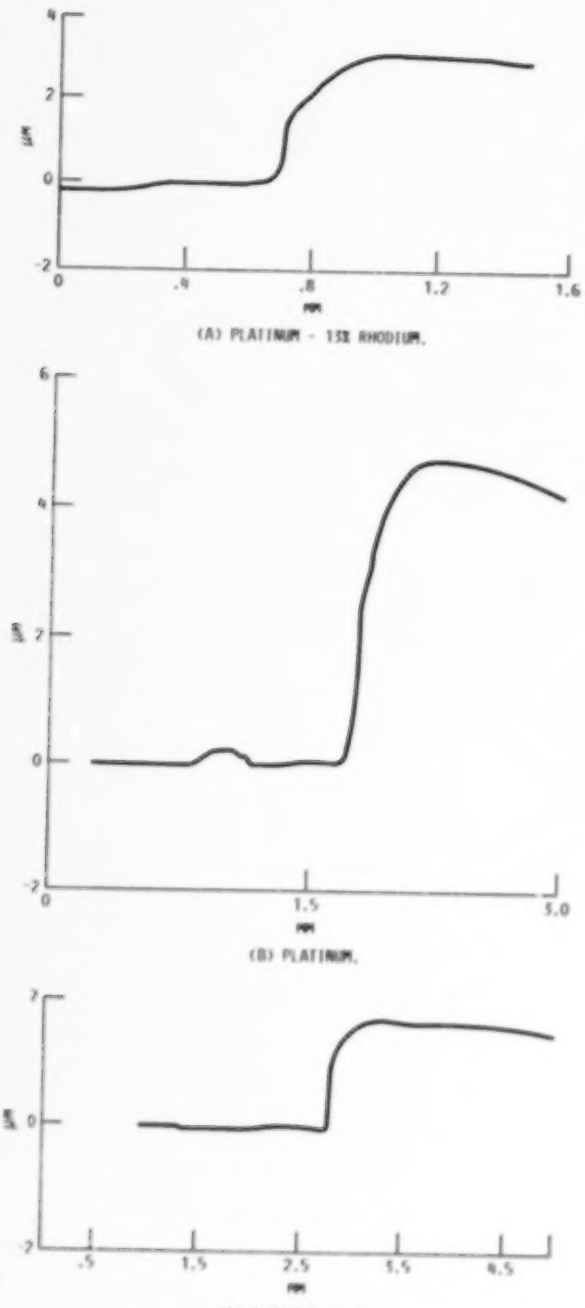


FIGURE 2. - PROFILOMETER TRACINGS OF THIN FILMS SPUTTER DEPOSITED ON GLASS SLIDES. PROFILE DISTANCE, mm VERSUS THICKNESS, μm.

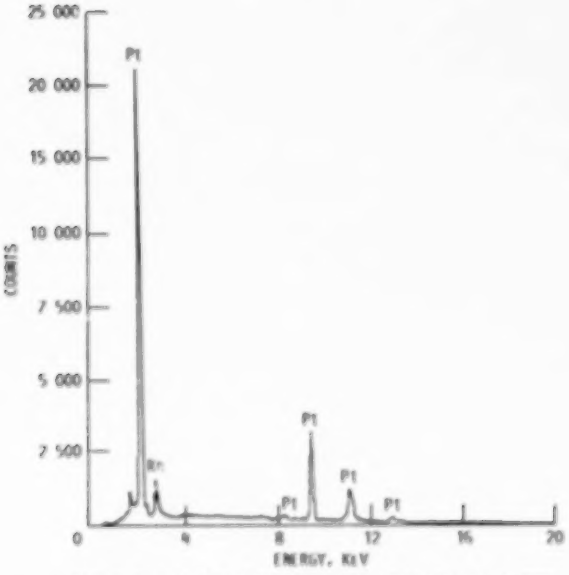
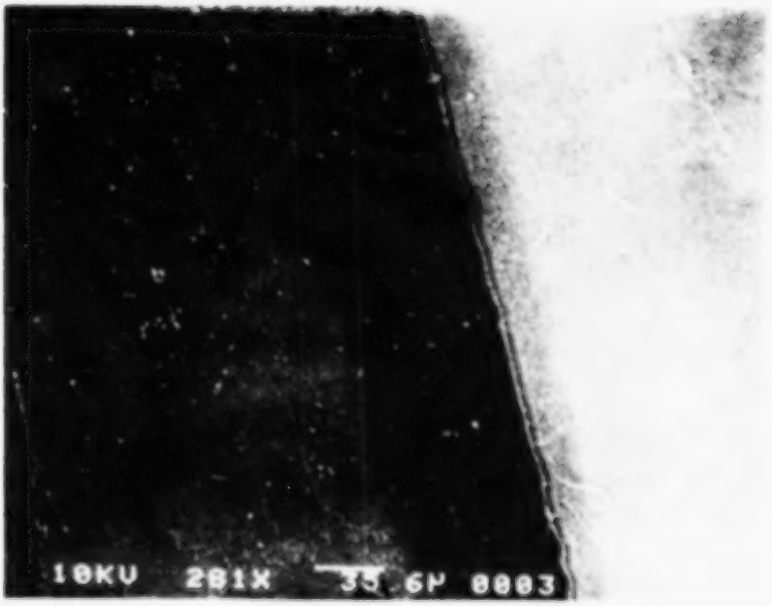
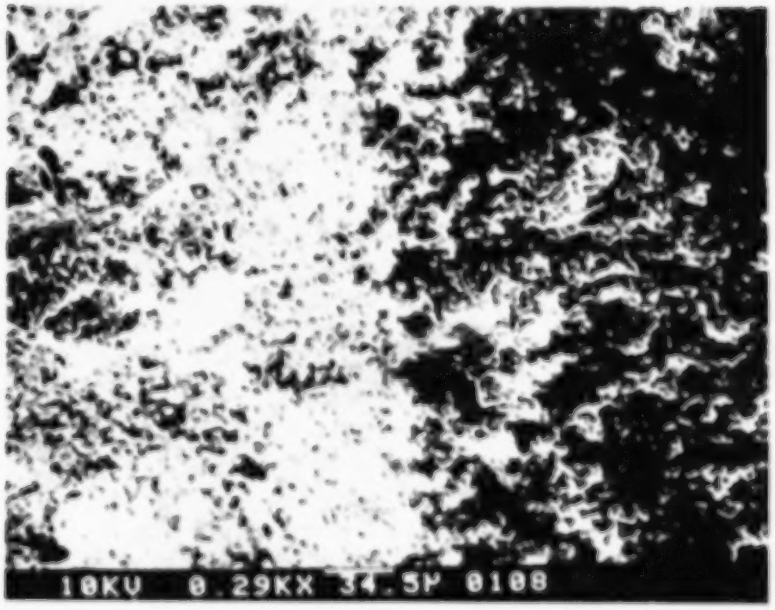


FIGURE 5. - ENERGY-DISPERSIVE X-RAY ANALYSIS OF PLATINUM - 15% RHODIUM FILM SPUTTERED ON THE PROTOTYPE SENSOR.





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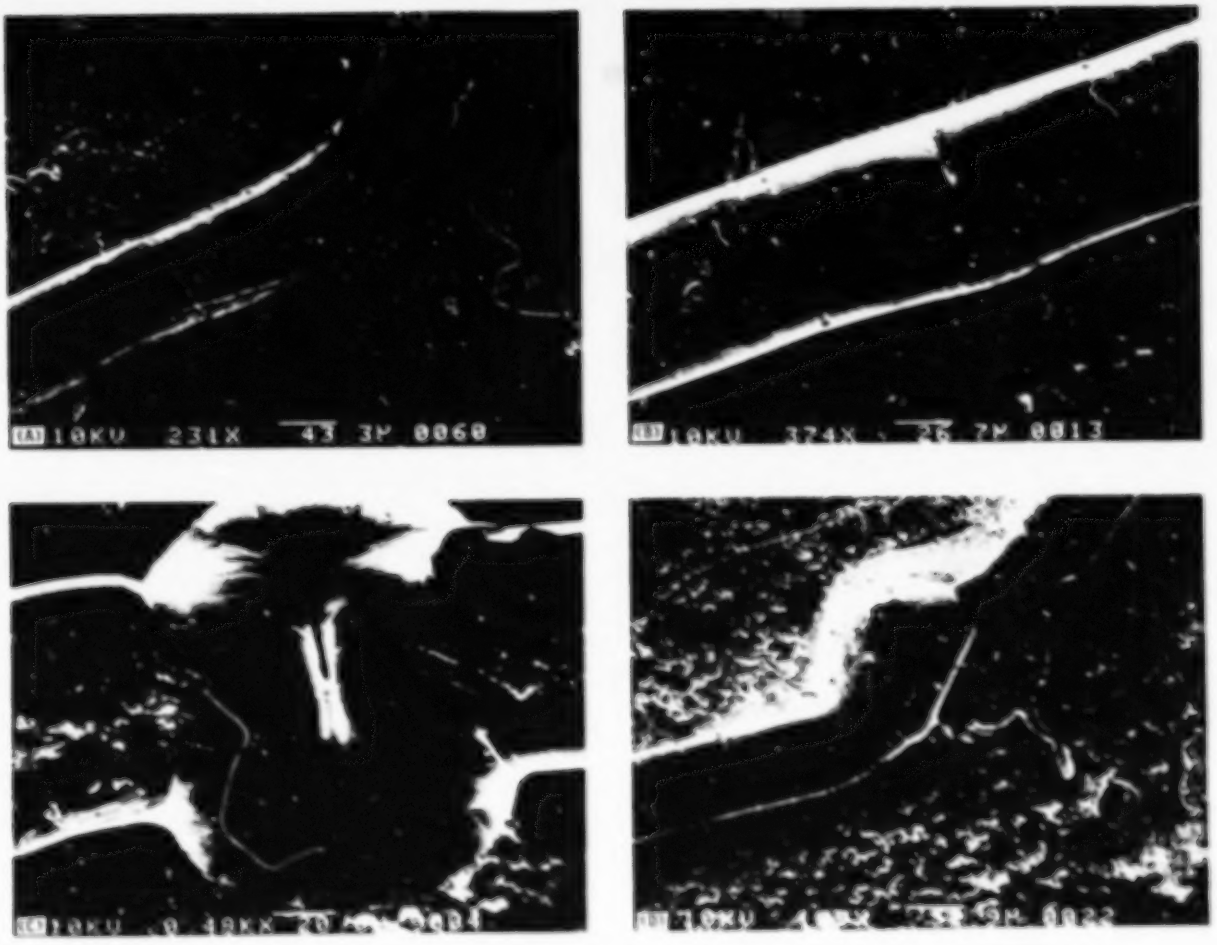

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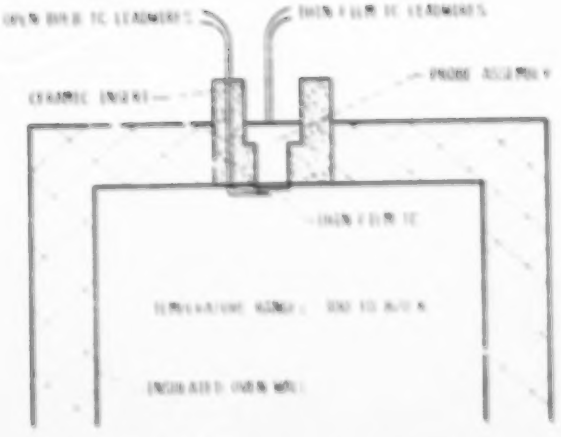

FIGURE 6. [illegible]

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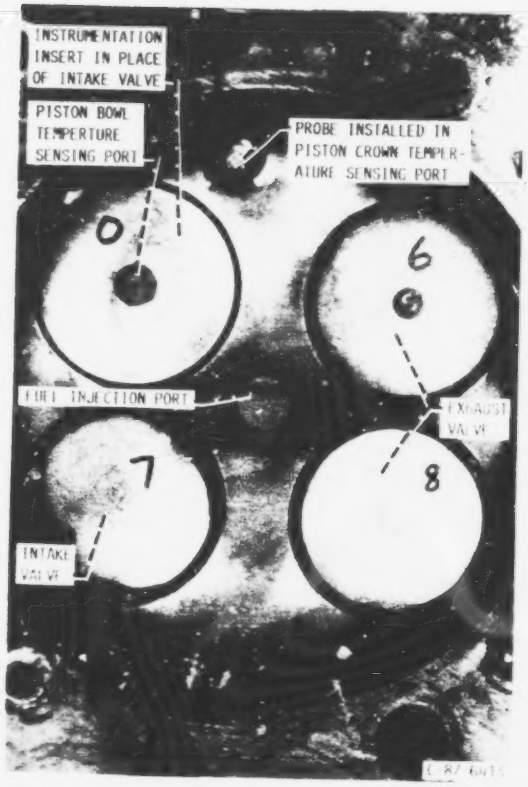


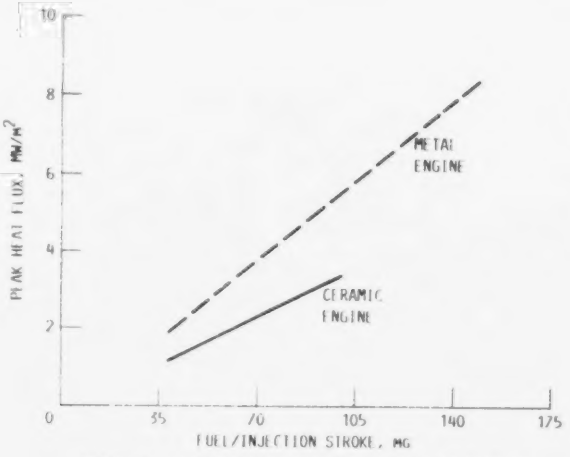
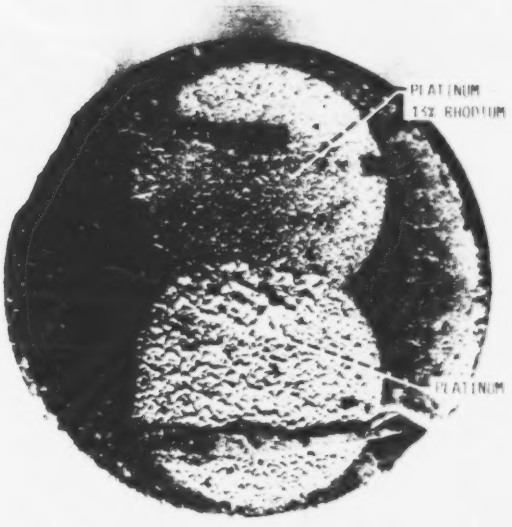


FIGURE 8. - COMPARISON OF MEASURED PEAK HEAT FLUX IN CERAMIC AND METAL ENGINES AT THE BOWL LOCATION.



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16. Abstract

A platinum versus platinum-13 percent rhodium thin-film thermocouple/heat-flux sensor was devised and tested in the harsh, high-temperature environment of a ceramic-insulated, low-heat-rejection diesel engine. The sensor probe assembly was developed to provide experimental validation of heat transfer and thermal analysis methodologies applicable to the insulated diesel engine concept. The thin-film thermocouple configuration was chosen to approximate an uninterrupted chamber surface and provide a one dimension heat-flux path through the probe body. The engine test was conducted by Purdue University for Integral Technologies Inc., under a DOE-funded contract managed by NASA Lewis Research Center. The thin-film sensor performed reliably during 6 to 10 hr of repeated engine runs at indicated mean surface temperatures up to 950 K. However, the sensor suffered partial loss of adhesion in the thin-film thermocouple junction area following maximum cyclic temperature excursions to greater than 1150 K.

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